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TESTS FOR NON-METALLIC PROTECTIVE COATINGS
FOR UNDERGROUND PIPES.

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I. INTRODUCTION

Although protective coatings have been applied for the protection of pipe lines for a great many years there are as yet no standard tests of coatings which can be used in the specification of a coating to be purchased. Because of this, coatings are being tested in a variety of ways. The purpose of this letter circular is to review briefly some of the tests more commonly applied to protective coatings and to point out some of the features of these methods.

The purpose of a test is to determine how the material under consideration will respond to certain forces or conditions which it may encounter in service. With respect to coatings intended to prevent corrosion of pipe exposed to soil, the destructive influences are moisture, changes in temperature, mechanical shock, and long-continued pressure or tension. No single test will determine the effects of all of these causes of failure, but since under some conditions one or another of these causes is the controlling factor, many tests have been developed which determine chiefly the effect of one destructive force.

For convenience the tests discussed in this paper have been subdivided into (1) laboratory tests of coated specimens, (2) tests of coating materials, and (3) field tests of coatings applied to short sections of pipe and to working lines.

II. LABORATORY TESTS FOR SPECIMENS OF COATINGS

1. Tests for the Absorption of Moisture.-

Since ferrous materials will not corrode in the absence of moisture, a coating which would permanently exclude moisture would prevent corrosion. Numerous tests have been devised to indicate whether a coating absorbs moisture. Three such tests are described by Thomas.¹

¹G.O.Thomas. The determination of the suitability of bituminous coatings for underground pipes. Jour. Inst. Eng., Australia, vol. 6, p.337, Oct. 1934.

It is sufficient to indicate the general nature of these tests. In one the bitumen is applied to bright steel plates 5 inches by 3 inches by 1/8 inch in size and the specimens are attached to a machine which alternately dips them in water and exposes them to the atmosphere. Other specimens are continuously submerged. The specimens are examined from time to time for a year or more for changes in color and surface condition. Finally, the specimens are weighed, the coating dissolved off, and the plates examined for rust stains. The test seems especially suitable for coatings to be used for lining water and sewer pipes. If coatings are to be compared they should be of the same thickness or of the thicknesses that would be used in practice. The results will be affected by temperature, character of the water, period of immersion, and the actinic quality of the light to which the specimens are exposed.

The second test consists of coating a piece of cleaned and weighed 26-gauge tin plate 5 inches by 32 inches in size with 10 g of the material to be tested. The coated sheet is reweighed. The edges of the specimen are protected by a pitch of known low water absorptiveness and again weighed. Specimens are then suspended in a tank of water kept in a dark dust-proof cupboard, the temperature of which is maintained at 60°F. After the required interval of time, usually 3 months, the specimens are removed, dried on the surface with a soft towel, and again weighed. The increase in weight is taken as the water absorption.

The third test described by Thomas is as follows. A quantity of the material under test is melted and poured into a form in which are placed bolts varying in length so that the

bolt heads lie at different distances (.018 to .08 inch) below the surface of the bitumen. Wires are brought out from the lower ends of the bolts and the upper surface of the bitumen is covered with water, care being taken to prevent water reaching the lead wires. The resistance between each of the bolts and the water is measured from time to time with a 500-volt Megger. In this way the progress of the penetration of moisture into the bitumen can be followed. The test requires a long period of exposure.

Another moisture absorption test has been described by Karl.²

² F.W.Karl. Discussion: Petroleum development and technology. 1926. A.I.M. & M.E., p.528. See also C.T.Langford, Accelerated corrosion tests, Proc.A.P.I. vol.X, No.2, p.93, Jan.3,1929.

A one-quart cardboard ice-cream carton is used as a container. The coating is applied to an 8-inch length of 1 1/4-inch iron pipe which is sealed to the bottom of the container by means of paraffin. A carbon electrode, such as is found in a No. 6 dry cell, is also placed in the container which is then filled with an electrolyte, such as water containing 1 percent of salt. Paraffin is then poured over the electrolyte to prevent evaporation and to hold the electrodes in place. Until moisture penetrates the coating there will be no difference of potential between the iron pipe and the carbon rod, but when moisture reaches the metal a difference of potential can be detected by a galvanometer or high-resistance, low-reading voltmeter. The amount of water absorbed is not shown by the deflection of the instrument but is indicated by the resistance of the circuit. This resistance can be measured accurately with an a-c bridge or roughly by a milliammeter and a voltmeter. If the milliammeter is used, a variable high resistance should be placed in series with the instrument and gradually cut out until a readable deflection is obtained. If the resistance of the coating is low the current will fall off as a result of polarization. Hence an instantaneous reading with an ammeter of suitable capacity and without a series resistance should be taken.

A somewhat similar test was used at one time by the Standard Oil Co. of California and has been described by Bly.³ The coating

³ E.P.Bly. Corrosion of underground pipe lines (Appendix). Petroleum Development and Technology, 1926, p.501, A.I.M. & M.E.

to be tested is applied to a steel plate upon which is placed a wooden frame coated with asphalt (a cylinder of glass was used in later tests) which was filled with sand moistened with a solution containing 70 g of NaCl and 30 g of Na₂SO₄ per liter. A steel plate slightly smaller than the insulating frame is placed upon the sand and held down by a clamp. Three such cells are made up for each coating. The coated plate is made anodic in one cell and cathodic in another by connecting a 6-volt storage battery

between the coated plate and the plate on top of the sand. No voltage is impressed on the third cell.

Lead wires are brought from each cell to a jack-type switch-board by means of which a large number of cells can be tested quickly. The resistance is at first measured every few hours, and later daily. Water is added to the cells from time to time to compensate for evaporation. The test is discontinued and the coated plates examined when the resistance of a cell reaches a constant value. This is an accelerated test depending upon the voltage and solution used as well as on the direction of the current. Although the impressed voltage may not be sufficiently high to hasten the initial breakdown of the coating, the complete failure of the coating is hastened by the current.

A somewhat similar test⁴ for coatings intended for the pre-
Burton McCollum and O.S.Peters. Surface insulation of pipes
as a means of preventing electrolysis. B.S.Tech.Paper 15, 1914.

vention of electrolysis was used by the National Bureau of Standards. Shallow cones of sheet iron were covered on the inside with the coating to be tested and the cone partly filled with water. Five cones were used for each coating. Two of the cones were connected to the positive pole of a 4-volt battery, one to the negative pole, and two carried no voltage. The circuits were completed by a few turns of copper wire dipping into the solution carried by the cones.

The resistance of each coating was measured by placing the cone in parallel with a 25,000-ohm resistance, measuring the joint resistance with a Wheatstone bridge, and calculating the resistance of the coating in ohms per square centimeter of coating surface exposed to the water. Precautions were taken to avoid leakage currents. The method does not permit a high degree of precision of measurement but was satisfactory for the purpose for which it was intended.

2. Tests for Resistance to Soil Stress.

All of the tests so far presented deal only with the absorption of moisture by a coating unaffected by other influences. Coatings exposed to soils undergo changes loosely described as weathering and are distorted by forces grouped under the term soil stress.

A laboratory test which takes account of soil stress as well as the absorption of moisture, used at the National Bureau of Standards to study the change in resistance of coatings has been described by Logan.⁵ The coating is applied to an 18-inch

⁵ K.H.Logan. Determining the value of pipe coating. Oil & Gas Jour. vol.27, No.50, p.33, May 2, 1929.

length of 1 1/2 to 2 1/2-inch pipe which is placed horizontally in the center of an insulated wooden box having internal dimensions of 10 inches in each direction. The ends of the pipe protrude through holes in the sides of the box just large enough to pass the coated pipe. The box is filled with soil having a large shrinkage factor. A carbon electrode is placed in one corner of the box. The soil is saturated with water, and the resistance between the coated pipe and the carbon rod is measured. A special instrument was designed for this measurement in order to use one instrument to cover a large range of resistances, but the measuring device is not an essential feature of the test. The soil is allowed to become thoroughly dry. It is then again saturated and the resistance measurement repeated, preferably a day or two after the addition of the water.

This test takes into account soil stress, but owing to the small amount of soil involved the stress is not as great as would be encountered in the field with the same soil conditions.

A more elaborate test involving soil stress was designed by Ewing⁶ who constructed of brick an outdoor soil box or tank which⁶ S. Ewing. Laboratory tests of new pipe coatings. Proc. AGA 1936. was filled with a heavy soil containing organic matter. To this soil was added 1 percent by weight of sodium carbonate in the form of a solution. This increased the shrinkage of the soil from 28.9 to 53.5 percent. Various coatings were applied to 2-foot sections of 2-inch steel pipe. The specimens were placed in the soil box and covered with 6 inches of soil which had been dried and reduced to clods about 1 inch in diameter. After two weeks the soil was thoroughly soaked. To duplicate field results it was found necessary to tamp the soil around the pipes when they were buried and again after it was wetted. Samples were exposed to one or two periods of wetting and drying covering periods of 4 and 6 months. The condition of the coating was determined by what is commonly referred to as the pattern test.

3. Pattern Test.

The pattern test depends upon the precipitation of ferrous ions, ferricyanide ions, or ferric ions by ferrocyanide ions. The coated pipe is first coated with a mixture of kaolin and water. Over this is spread the pattern paper which is then covered by a wet outing-flannel pad. The pad is held in place by a saddle consisting of four half-inch strips of copper equally spaced 4 inches apart by insulating strips. The copper strips are sufficiently long to nearly encircle the pad-covered pipe and are held in position by a heavy cord laced between lugs on the ends of the saddle. A battery is connected between the pipe and the copper strips. The procedure recommended by Ewing and

Scott⁷ is as follows:

⁷ Scott P. Ewing and Gordon N. Scott. Electrolytic method for determining condition of non-metallic pipe coating. American Gas Association Monthly, vol. XVI, No. 4, p.137 (1934).

(1) The clay should be composed of kaolin thoroughly mixed with 1 1/2 times its weight of water, slightly acid to litmus, and its resistivity should be not less than 1000 ohm-cm.

Most tap waters will be found suitable for mixing the clay. If the water used is good to drink, it may be assumed without adjustment or test that the pH and resistivity are within acceptable limits. However, if the paper develops a uniform faint blue stain (acid water) or if the patterns appear to be weak (alkaline water) it may be necessary to adjust the pH of the water with either ammonia or acetic acid (vinegar). The clay should be mixed and used in enameled buckets or stone crocks to prevent contamination from rust. Kaolin may be obtained from dealers in ceramic materials.

(2) The developer consists of a 1 percent solution of potassium ferricyanide (2 tablespoonfuls of the salt per gallon of water) made acid to litmus with acetic acid.

The developer should also be used in stone crocks or enameled buckets.

(3) The pad solution consists of ordinary tap water (adjusted as in paragraph (1) if necessary) to which has been added two tablespoonfuls per gallon of the developer described in paragraph (2).

The addition of the developer to the pad solution prevents any ferrous or ferric ions or rust stains carried to the pad in a previous test from spuriously staining fresh pattern paper. Fresh pad solutions should be made up from time to time. This solution should likewise be used in stone crocks or enameled buckets.

(4) One linear foot of the coated pipe surface should be tested.

Field experience indicates that this is a sufficiently large and convenient sample of the coating for test.

(5) The surface of the coating to be tested should be clean and free from adhering soil.

Since the presence of colloidal soils in crevices, craters or other distorted areas tends to prevent patterns of maximum density the coating must be freed of all adhering soil. Vigorous brushing with an ordinary fiber scrub brush and plenty of clean water will remove the most tenaciously adhesive clays and not damage the coating.

(6) The clay should be well brushed into the coating on the selected linear foot and smeared on thick enough to cover the entire coating.

Especial care should be exercised to see that the clay is worked into cracks, crevices, air holes or other small discontinuities in the coating.

(7) The pattern paper should be wrapped tightly over the moist clay.

The pattern paper should be tough, readily absorptive of moisture and preferably creped, so that it can be stretched where necessary over protruding irregularities in the coating. The moisture absorbing property of the paper may be considered satisfactory if an appreciable current passes through it in 15 seconds when a piece of the paper, laid on a metallic surface and a moist pad about one square inch in area pressed against it, is subjected to the potential of an ordinary dry cell. A "pinhole crepe paper," manufactured by the Perkins Goodwin Co., 551-5th Ave., New York City, satisfies the above requirements. The pattern paper should be twelve inches wide and sufficiently long to wrap around the pipe and lap two to three inches. It is convenient to have the pattern paper in the form of a loose roll. The free edge should be pressed into the moist clay parallel to the axis of the pipe and the paper unrolled around the pipe while one hand smooths the paper pressing it onto the clay. All air pockets should be expelled. If the clay has the proper amount of moisture in it, the pattern paper will soon become moist. The paper should be further moistened and smoothed by folding a wet pad so that a conveniently small flat surface may be used to "iron" the paper. If the pattern is to be used in locating the discontinuities in the coating it should of course be applied to the pipe in a specified manner so that it can be replaced in its original position on the pipe after it has been dried. If the pattern is to serve as a permanent record it may be appropriately marked with an indelible pencil before it is applied to the coating.

(8) The pad should then be wrapped tightly over the pattern paper.

The pad, which is 12 inches in width, consists of at least three thicknesses of a heavy grade of outing flannel folded from 36-inch width material. When not in use during the course of a series of tests the pad should be kept in the pad solution.

(9) A saddle is then wrapped and tied not too tightly over the pad to serve as an electrode to which electrical contacts may be easily made.

The saddle has been previously described. Copper strips should be used in the saddle assembly.

(10) Pad solution is then poured over complete set-up until water starts to drip from the under side of the pad.

(11) A potential is applied between the pipe and saddle, the pipe being anodic. The product of the applied potential in volts and the time of application in minutes is approximately, and not less than, sixty.

A radio "B" battery, several dry cells in series or an automobile storage battery will serve as a convenient source of current. Since conductance tests are usually made in conjunction with the pattern tests the conductance of the coating should be measured before the application of the potential.

(12) The "set-up" is then taken down, the pad returned to the pad solution and the pattern paper carefully unwrapped and immersed in the developer.

The pattern paper should be carefully immersed in the solution so that no part of the paper touches any other part before it reaches the developing solution. Careful immersion avoids the appearance of spurious stains where no rupture occurs in the coating.

(13) The pattern paper should be left in the developer for not less than one minute and should be occasionally agitated after which it should be thoroughly washed in clean water and dried.

4. Conductance Test.

The conductance measurement referred to in section (11) is made with a voltmeter and milliammeter. A special instrument⁸

⁸ E.R. Shepard. Measurement of electrical conductance of non-metallic pipe coatings. Am. Gas Jour. 136, p.22, June 1932. This instrument can be purchased from The Sensitive Research Instrument Co., 4545 Bronx Boulevard, New York, N.Y.

has been designed for this work. The instrument saves considerable time when a large number of measurements are to be made, but any suitable set of instruments will produce the same results. A low-reading, high-resistance voltmeter is required.

The density of the patterns in the pattern test and the apparent conductance of the coating are both affected by the conductivity of the solutions used to wet the coating.

The pattern test has been applied to all of the coatings removed from the American Gas Association and American Petroleum Institute test sites.

III. TESTS OF COATING MATERIALS

Most of the tests so far discussed are tests of coatings as a whole, and require a relatively long period of exposure to determine the performance of the coating. Certain laboratory tests yield data from which this performance can be estimated. Some of these are listed below.

1. Percentage and Size of Filling Materials.

The amount and character of the material added to asphalt or coal-tar pitch to form an enamel influences the resistance of the enamel to soil stress. Specific data as to the most desirable size, amount, and kind of filler are not available but the amount of the filler can be determined approximately by ignition and its fineness by sieve tests.

2. Laboratory Performance Tests.

The tendency of a coating to flow is roughly related to softening point. This characteristic can be determined by A.S.T.M. Standard Test for Softening Point D36-26.

The resistance of a coating material to pressure is indicated in a general way by the penetration test. A recognized method for making this test will be found in A.S.T.M. Test for Penetration of Bituminous Materials D5-25. The softening point and penetration tests are also described in Federal Specification SS-A-666, Asphalt for roofing, waterproofing and dampproofing, copies of which can be obtained from the Superintendent of Documents, Washington, D.C., price 5 cents. The penetration test is not satisfactory for enamels containing particles of large diameter. A test described as being more suitable for this type of enamel has been proposed by a manufacturer of this type of enamel.⁹

⁹ Pressure deformation of bituminous pipe coating materials. Technical Sales Bulletin TS-3-32. The Barrett Co., New York.

The following instructions for testing coating materials are taken from a set of specifications for the purchase of a bitumen coated steel water pipe.

"D-2. Testing.

D-2a. The primer and enamel shall be applied to clean, soft, steel plates (10 by 12 by $\frac{3}{22}$ inches), leaving a narrow uncoated border about the edge of the plate; after the primer has been allowed to dry 72 hours the enamel shall be applied to a thickness between $\frac{1}{16}$ -inch and $\frac{3}{22}$ -inch by pouring over the plate. The spreading and working qualities, together with the character of the resultant surface, shall in general determine workability.

D-2b. The plates used for the test prescribed in paragraph D-2a shall have lines 2 inches apart drawn across the face of the enamel and to the edge of the plate. The plate so prepared shall be suspended for 5 hours in a vertical position in an oven or heating chamber which is maintained at a constant temperature of 140°F. At the end of 5 hours the plate shall be examined for sagging of the enamel as indicated by shifting of the lines drawn across the coating and plate.

D-2c. Susceptibility to cracking at low-air temperatures shall be determined by placing the sample which was used for the heat test in a refrigerated chamber in which the air temperature is maintained at 0°F plus or minus 5°F for 5 hours. At the end of 5 hours the plate shall be examined for cracking, checking, or peeling, removed, allowed to remain at room temperature for 2 hours, and examined again.

D-2d. Brittleness shall be determined on the samples which were used in the two foregoing tests after the coated plates have been allowed to stand at room temperature (77°F to 85°F) at least 2 hours. The first plate shall be supported by a block on the floor so as to give support beneath the point of impact of the falling ball. A 750 g steel ball shall be dropped from a height of 6 feet on the coating at a point at least 4 inches from the edge of the plate. Evidence of shattering of the coating from the plate or radial cracks from the center of the point of impact shall be looked for. The second plate shall then be laid, coating downward, on a board through which a hole about $3\frac{1}{2}$ inches in diameter has been cut. The same ball shall be dropped from a height of 6 feet and shall strike the plate over the center of the hole in the board supporting the plate. Any evidence of shattering of coating or the development of cracks shall be looked for.

D-2e. Penetrations shall be run in accordance with the A.S.T.M. method D-5-25; the samples of the enamel made up from the composites obtained as prescribed above. Three standard penetration samples shall be made up and tested at 115°F, 77°F, and 32°F; 5 penetrations to be taken at each temperature and the results of each temperature averaged. The samples from the 77°F and 115°F tests shall then be placed in an oven or heating chamber and subjected for 5 hours to a constant temperature of 200°F. The samples shall then be removed, melted at the lowest possible temperature, thoroughly stirred, and again tested at 77°F and 115°F; 5 penetrations to be taken at each temperature and the results averaged as before."

The result of the flow test will depend somewhat on the smoothness of the plates to which the coating is applied. The smoother the surface the more severe the test.

IV. FIELD TESTS

1. Exposure Tests.

There are two classes of field tests of protective coatings. One class involves the shop application of the coating to short sections of small-diameter pipe and the subsequent burial of the coated pipe at suitable test sites. After a period of one or more years the coatings are examined, preferably by some such method as that used by Ewing and Scott,¹⁰ together with the de-

¹⁰

see footnote 7

termination of the condition of the pipe beneath the coating. The depths of the pits should be measured with a depth gage - not guessed at. Because all field corrosion data are erratic, several specimens of the same coating should be tested.

This method is suitable for the testing of a large group of coatings at one time. The test is not as severe as the application of the coating to a working pipe line. The latter is the type of test which gives the most reliable results when conducted on a sufficiently large scale. The objectionable features are the cost of the test and the difficulty in securing suitable lines and test locations. Both of the above-mentioned field tests should extend over long periods since the deterioration of most coatings is progressive, and satisfactory coatings must prevent corrosion for long periods.

2. Test for Continuity.

Poor application is a frequent cause of coating failures. Inspections do not always disclose faulty applications of coatings. A much surer method for determining the continuity of

coatings as well as for locating bare spots has been developed by Clarvoo.¹¹

¹² G.W. Clarvoo: The detection of flaws in pipe line protective coatings before burial. Pipe Line News, V, No. 8, p. 13, July 1933.

The test consists of applying a suitably high potential across the coating by connecting one side of an induction coil to a bare spot on the pipe while the other terminal is connected to a metallic brush which is passed slowly over the surface of the coating. Some users have attached the brush to the outer or inner circumference of a wooden ring which is hauled through or over the coated pipe depending on whether an inside or an exterior coating is to be tested. The voltage should be adjusted according to the thickness of the coating and should not be allowed to remain across a single point for more than a few seconds. Too high a voltage or application for too long a time may injure the coating. The test has been used successfully on many occasions to locate flaws which would have been overlooked otherwise. The test is usually made at the point where the coating is applied and there is a possibility that the coating may be injured subsequently when the coated pipe is transported or laid. The simplest way of making this type of injury evident is to white-wash the coated pipe or to wrap paper around it so that an abrasion will be easily noticed on account of the exposure of the black coating through the white or light-colored cover coat.

An additional test for the continuity of the coating after the pipe line has been completed consists of subdividing the line by insulating joints and measuring the resistance between the pipe line and a remote ground by means of an insulated lead wire attached to each section of pipe. A test of this kind has been conducted by Kuhn.¹²

¹² R.J. Kuhn. Testing pipe line coatings in place. Oil & Gas Jour. vol. 30, No. 49, p. 47.

Another method of testing coated lines without exposing them is described by an anonymous author.¹³ The instrument used con-

¹³ Ingenious instrument detects and traces buried pipe. Oil & Gas Jour. vol. 32, No. 43, p. 71, March 15, 1934.

sists of an induction coil and a loop antenna connected to a vacuum-tube amplifier. It was originally designed as a pipe locator. The secondary of the induction coil is connected between the pipe and a remote ground. At points where there is increased leakage on account of a bare or weak place in the coating, the sound in the ear phones connected to the amplifier becomes louder.